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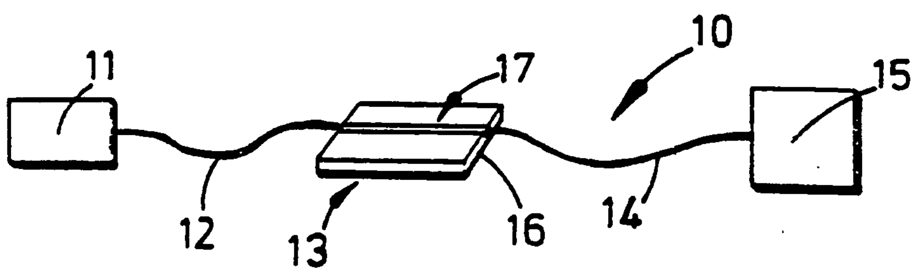

(57) Abstract

A guided optical wave chemical sensor system (10) is adapted to measure the concentration of a chemical species in the presence of thin film surface contaminants on the waveguide (17). The system (10) has a light source (11) which launches tw different optical waves into the waveguide (17) to establish evanescent fields (24, 25) of different penetrati n depths. The detector (15), which includes a data store and an arithmetic unit, is pre-calibrated using a known concentration of the chemical species and the same two different ptical waves but prior to the waveguide (17) having been exposed to surface contaminants. When surface contaminati n is present the detector (15) evaluates the thickness f the contaminant layer from the pre-calibration data and hence evaluates the (unknown) concentration f the chemical species according to a predetermined alg rithm.

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GUIDED OPTICAL WAVE CHEMICAL SENSOR SYSTEMS

This invention relates to guided optical wave chemical sensor systems.

Guided wave chemical sensor systems are known and comprise a waveguide sensor device coupled via one or more waveguides to a light source and a light detector. The sensor device comprises a substrate supporting an optical waveguide and, is constructed so that, when it is in use, the optical field has an evanescent portion which penetrates into the medium adjacent the sensor device as a result of which there is interaction with the chemical characteristics of the medium and this interaction manifests itself at the detector. Accordingly changes in the chemical characteristics produce changes in the detector reading.

The known sensor devices have proved very difficult to use particularly when the medium is gaseous and the system has been intended to identify the occurrence of a gaseous species. It is believed that this is due to relatively low concentration of the species at the sensor device, for example due to gaseous dispersion, or to inadequate sensitivity of the sensor device, or due to contamination of the sensor device.

It is an object of the present invention to provide a new and improved guided wave chemical sensor system.

According to the present invention there is provided a guided wave evanescent field chemical sensor system comprising a sensor device having a substrate supporting an optical waveguide and optically coupled between a light source and a light detector, wherein the light source is adapted to launch two different optical waves into the sensor device to establish evanescent fields of different penetration depths, and the detector is adapted to measure the sensor devic outputs arising from said different

waves and to evaluate the concentration of the chemical species according to a predetermined algorithm.

By virtue of the present invention the sensitivity of the device is rendered independent of thin film surface contaminants on the waveguide so that an accurate measurement can be taken of the chemical species. By 'thin film' we mean films which have a thickness of or less than the order of magnitude of the optical wavelength emergent from the light source.

For aqueous and gaseous media where the refractive index is typically in the range 1.00 to 1.33 highest sensitivity is attained in practice with a sensor device having a substrate with a refractive index as close as possible to that of the medium (and, of course, the refractive index of the waveguide should be larger). Thickness of the waveguide is also a factor that affects sensitivity. The waveguide may be embedded in the substrate or be of the ridge type, i.e., surface mounted on the substrate, the latter having greater sensitivity than the former.

Also it is preferred that the waveguide be of the step index type rather than of the graded index type since the former usually provides more sensitivity than the latter.

The waveguides may be, or form part of, an optical resonator. Thus, the resonator may be formed as a Fabry-Perot cavity incorporating the waveguide or as a Bragg resonator incorporating the waveguide, both of which produce standing waves along the length of the waveguide. Alternatively, the resonator may be formed as a ring or loop secondary waveguide coupled at one point around its circumference to a principal waveguide which gives rise to a travelling wave in the secondary waveguide which is adapted to produce an evanescent field for penetrating the adjacent medium. The principal waveguide may also be adapted to produce an evanescent field.

Alt rnatively, the waveguide may simply be a principal waveguide which is free of optical resonance.

Further, the present invention provides a method of measuring the concentration of a chemical species using the system of the present invention and in the presence of thin-film surface contaminants on the waveguide, comprising effecting a pre-calibration of the system in the presence of a known concentration (C_o) of the chemical species prior to the waveguide being exposed to any surface contaminant, the pre-calibration comprising

- (a) launching into the waveguide the said two different optical waves;
- (b) monitoring the respective outputs (D_1,D_2) from the detector;
- (c) evaluating respective concentration - proportionality constants (K_1K_2) from the formula $D = K.C_o$
- (d) measuring the respective evanescent field penetration depths (d_1d_2)
- (e) storing the parameters d_1 d_2 K_1 K_2

and - (f) arranging the detector sequentially to evaluate the equations

$$t = [d_1d_2/2(d_1-d_2)].\ \log_n[K_2D_1'/K_1D_2']$$

and $$C' = D_1'/K_1\exp(-2t/d_1)$$

in respect of subsequent detector outputs D_1' and D_2' arising from the subsequent launching of said two different optical waves into the waveguide having a thin film surface contaminant of thickness t and in the presence of a concentration C′ of said chemical species and which is to be measured.

Embodiments of the present invention will now be describ d by way of example with r fer nc to the

accompanying drawings, in which:

Fig. 1 schematically illustrates a guided wave sensor system;

Figs. 2, 3 and 4 illustrate different forms of sensor device for use in the Fig. 1 system;

Fig. 5 illustrates typical response characteristics of the Fig. 4 device;

Fig. 6 illustrates the evanescent fields of two different optical waves propagating along a waveguide of the Fig. 1 system in accordance with the present invention; and

Fig. 7 illustrates a modified form of a Fig. 6 waveguide.

The guided wave sensor system 10 shown in Fig. 1 comprises a light source 11 coupled via a waveguide 12 to a sensor device 13 the output of which is coupled via a waveguide 14 to a detector 15. The source 11 provides light output for the sensor device 13 at a wavelength which is appropriate to the medium to be sensed and typically is provided by a laser, preferably tunable, or a light emitting diode (LED) operating at an accurately stabilised wavelength. An alternative form of light source is an incandescent bulb with associated spectrometer which provides a less restricted range of wavelengths but imposes difficulties in launching the light into the guide 12. Guides 12, 14, are typically optical fibres and the detector 15 may be a colour sensitive diode or, a dichroic or absorption filter with matched detection elements which are wavelength sensitive. Detector 15 and guide 14 may be located on the same side of sensor device 13 as the source 11 if a reflector is mounted at the end of device 13 remote from source 11 and a beam splitter is used to couple guide 14 to wavelength 12.

Sensor device 13 may alternatively take any one of the

forms particularly illustrated in Figs. 2, 3 and 4 in each of which a substrate 16 supports a waveguide 17 forming part of or being coupled to a resonator 20.

In Fig. 2 the resonator 20 is of the Fabry-Perot type provided by mirrors 22A, 22B, preferably dielectric mirrors, at each end of the waveguide 17 so that according to the length of the waveguide 17 a standing resonance wave is established in the waveguide 17 when the source 11 is tuned to the resonant frequency of the resonator 20.

In Fig. 3 the resonator 20 is of the Bragg type provided by a distributed reflective transverse grating 23 coated onto the waveguide 17 along its length so that, like the Fig. 2 arrangement, a standing resonance wave is established in the waveguide 17. The grating 23 need only exist at the two ends of waveguide 17 to operate as a resonator.

In Fig. 4 the resonator 20 is of the ring or loop type separate from the waveguide 17 but coupled thereto at one point around its circumference and which gives rise to a travelling resonance wave in the loop guide which has the form illustrated at A in Fig. 5, the spacing Δf between peaks being a function of the loop length (ℓ) and the effective refractive index of the waveguide. The presence of the medium in contact with the sensor device 13 attenuates the amplitude of the resonance wave A to wave B shown also in Fig. 5, so that the finesse of the resonator 20, represented by the ratio $\Delta f/\delta f$, is a measure of the losses in the medium, and therefore a measure of the chemical characteristics of the medium.

In each resonator form the response characteristics take the same form as Fig. 5 but with different scaling and the resonant wave may be tailored so that the resonant spacing Δf matches the absorption characteristics of the chemical species being sensed for the purposes of enhancing sensitivity. For example, if the chemical

species is methane (CH_4) which is known to have an absorption spectrum in the 1.66 micron band consisting of a series of nearly-equally spaced lines about 60 GHz apart the resonator can be designed so that Δf = 60 GHz by selecting the loop length for a given waveguide refractive index.

During the use of the sensor device 13, a thin contaminant layer may form on the surface of the waveguide 17. The contaminant layer may be, for example, a thin (~ 100nm) layer of water, oil or some other substance. The presence of thin layer surface contaminants modifies the output of the device 13 so that it is a function of contaminant layer thickness (measured in fractions of a wavelength) and the concentration of the chemical species. However, in accordance with the present invention, there are various ways of correcting the measurements when such surface contaminants are present, now explained with reference to Figs. 6 and 7.

In Fig. 6, 24 and 25 are the evanescent fields of two different optical waves delivered by source 11 and supported either simultaneously or otherwise by waveguide 17. The two optical waves are chosen so that the penetration depth of evanescent field 25 into the chemical species is greater than that of field 24. As is well known the penetration depth of the evanescent field is related to the propagation constant of the optical wave in the waveguide 17 and, for thin contaminant layers, is independent of the nature of any external contaminant. Examples of pairs of optical waves which have different penetration depths and which may be used in accordance with the present invention are:

(i) The zeroth-order and first-order modes of the waveguide 17;

(ii) Two waves of different wavelengths supported by waveguide 17 and which correspond to absorption

lines of the chemical species, e.g., wavelengths of 1.33 and 1.66 microns for methane; and

(iii) Two waves of different polarisation supported by waveguide 17, specifically the transverse electric (TE) and the transverse magnetic (TM) modes.

In order to compensate for surface contamination a ratiometric measurement is taken at the output of the detector 15 for the two optical waves. For sensor device 13, it is known that, with no contamination present, and for a particular optical wave, the detector reading, D, (after suitable electronic processing) is proportional to the concentration C of the chemical species, i.e.,

$$D = K.C \qquad (1)$$

where K is a constant determined by calibration of the sensor device for the optical wave.

When contamination is present, the detector output decreases exponentially with the (unknown) contaminant layer thickness t i.e.,

$$D = K.C.\ \exp\ [-\ 2t/d] \qquad (2)$$

where d is the penetration depth of the evanescent field of the optical wave, the value of which is also obtained during the calibration of the sensor device for the optical wave.

The penetration depth d is obtained by evaluation of the known formula:

$$d = \lambda(n_e^2-n_r^2)^{\frac{1}{2}}./2\pi$$

where λ is the wavelength of the optical wave;

n_r is the refractive index (real part) f the

chemical species ($n_r = 1$ for gases);

n_e is the effective index of the optical wave and is measured by m-line spectroscopy using the prism-coupler method described by Ulrich and Torge in "measurement of thin film parameters with a prism coupler" published in Applied Optics Vol.12 (1973) at pages 2901-2908.

With two different optical waves, two detector readings are obtained such that:

$$D_1=K_1.C.\exp[-2t/d_1] \quad (3)$$
$$D_2=K_2.C.\exp[-2t/d_2] \quad (4)$$

From equations (3) and (4) we obtain the thickness of the contaminant layer as:

$$t=[d_1d_2/2(d_1-d_2)].\log_n[K_2D_1/K_1D_2] \quad (5)$$

Hence t may be calculated from the measured ratio of D_1/D_2 and the other (known) constants of the sensor device. This value of t can then be used in either equation (3) or (4) to obtain the correct measure of concentration C of the chemical species.

In order to further improve the sensitivity of the sensor device 13 and to improve the accuracy of the compensation for surface contamination, a composite waveguide (also known as a 4-layer waveguide) may be used as illustrated in Fig. 7. In Fig. 7, 26 is a thin (100-500 nm) film of high refractive index ($n \geqslant 2$) material. The ends 27, 28 of the film are tapered in thickness. Typical materials for 26 are titanium dioxide (TiO_2), silicon nitride (Si_3N_4), niobium pentoxide (Nb_2O_5), tantalum pentoxide (Ta_2O_5) or arsenic trisulphide (As_2S_3). Depending on the thickness of the film 26, optical waves launched at 12 into waveguide 17 may be

transferred in whole or in part into the overlay film 26 at taper 27. After travelling along the overlay film 26, the optical wave is coupled back to guide 17 at taper 28. The sensitivity of sensor device 13 may thereby be substantially improved, since the presence of a thin high refractive index layer on the surface effectively increases the field level at the surface and thereby increases the magnitude of the evanescent tail. Unpolarised light may be launched into guide 17 and the output passed through a polariser device 29. The output at 30 is then measured by detector 15 for two angular positions of the polariser device 29 (corresponding to polarisation directions parallel and perpendicular to the waveguide surface) and the ratio of the measurements may then be used to compensate for surface contamination as previously described.

It will be appreciated that to fulfil the requirements of the present invention, the detector 15 requires an arithmetic unit and data store to enable evaluation of equation 5 from stored values of d_1, d_2, K_1, K_2 and the stored ratio D_1/D_2. The arithmetic unit therefore evaluates concentration $\underline{C}$ of the chemical species from equation 3 or equation 4 using the value of $\underline{t}$ evaluated from equation 5.

CLAIMS:

1. A guided wave evanescent field chemical sensor system comprising a sensor device having a substrate supporting an optical waveguide and optically coupled between a light source and a light detector, wherein the light source is adapted to launch two different optical waves into the sensor device to establish evanescent fields of different penetration depths, and the detector is adapted to measure the sensor device outputs arising from said different waves and to evaluate the concentration of the chemical species according to a predetermined algorithm.

2. A system as claimed in Claim 1, wherein the two different optical waves are the zeroth-order and the first order modes of the optical waveguide.

3. A system as claimed in Claim 1, wherein the two different optical waves are of differing wavelength corresponding to absorption lines of the chemical species and which wavelengths are supported by the optical waveguide.

4. A system as claimed in Claim 1, wherein the two different optical waves are of differing polarisation.

5. A system as claimed in any preceding claim, wherein the two different optical waves are launched contemporaneously into the optical waveguide.

6. A system as claimed in any one of Claims 1-4, wherein the two different optical waves are launched consecutively into the optical waveguide.

7. A method of measuring the concentration of a chemical species using the system of the present invention according to Claim 1 and in the presence of thin-film surface contaminants on the waveguide comprising effecting a pre-calibration of the system in the presence of a known concentration (C_o) of the chemical species prior to the waveguide being exposed to any surface contaminant, the

pre-calibration comprising

- (a) launching into the waveguide the said two different optical waves;
- (b) monitoring the respective outputs (D_1, D_2) from the detector;
- (c) evaluating respective concentration - proportionality constants (K_1K_2) from the formula $D = K.C_o$
- (d) measuring the respective evanescent field penetration depths (d_1d_2)
- (e) storing the parameters d_1 d_2 K_1 K_2

and - (f) arranging the detector sequentially to evaluate the equations

$$t = [d_1d_2/2(d_1-d_2)].\ \log_n[K_2D'_1/K_1D'_2]$$

and

$$C' = D'_1/K_1\exp(-2t/d_1)$$

in respect of subsequent detector outputs D'_1 and D'_2 arising from the subsequent launching of said two different optical waves into the waveguide having a thin film surface contaminant of thickness t and in the presence of a concentration C′ of said chemical species and which is to be measured.

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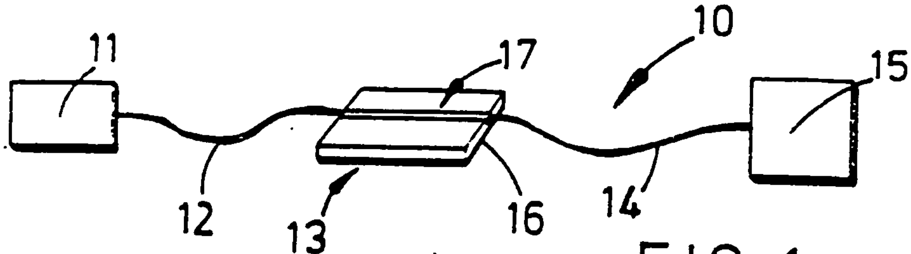

FIG.1

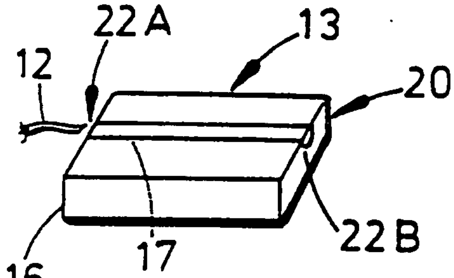

FIG.2

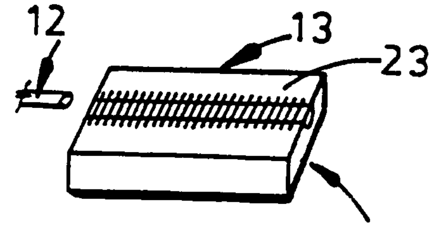

FIG 3

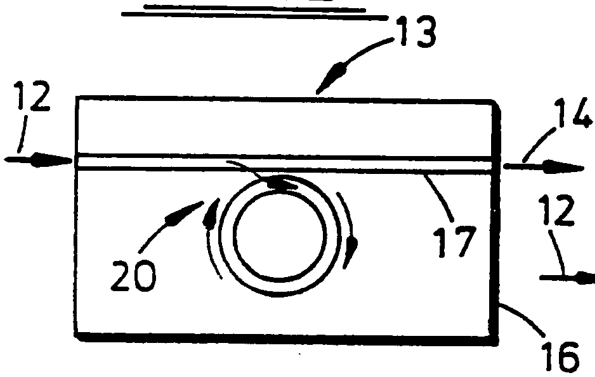

FIG.4

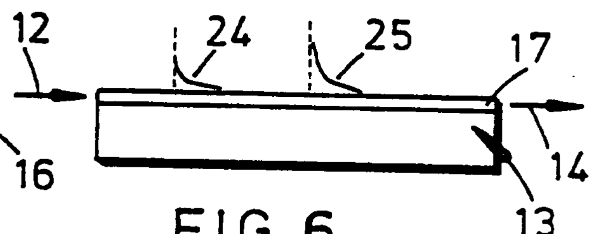

FIG.6

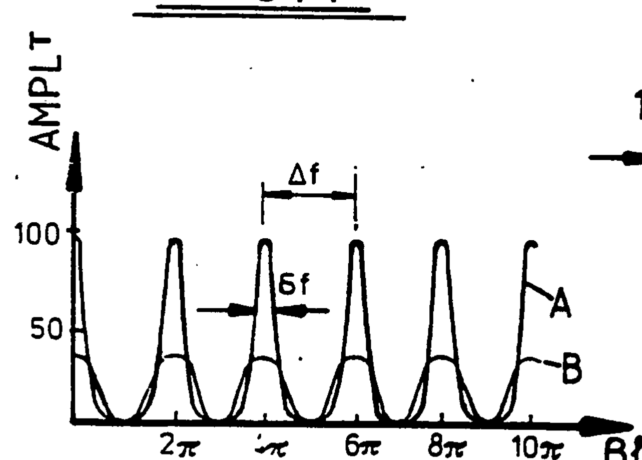

FIG.5

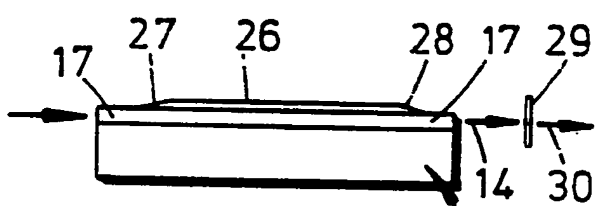

FIG.7

INTERNATIONAL SEARCH REPORT

International Application No PCT/GB 90/01332

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC^5: G 01 N 21/45

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
IPC^5	G 01 N 21/45, G 01 N 21/43, G 01 N 21/55

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category [*]	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No. [13]
X	Applied Physics Letters, volume 52, no. 10, 7 March 1988, American Institute of Physics, (New York, NY, US) R. Reuter et al.: "Monitoring humidity by polyimide lightguides", pages 778-779 see the whole article --	1,4,5
X	WO, A, 89/07756 (W. LUKOSZ) 24 August 1989 see pages 6-7; page 14, figure 4; pages 15-16 --	1,4,5,7
A	IBM Technical Disclosure Bulletin, volume 29, no. 8, January 1987, IBM Corp., "Fiber-optic analysis device", page 3309 see the whole article --	1

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IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
6th December 1990	19 DEC 1990
International Searching Authority	**Signature of Authorized Officer**
EUROPEAN PATENT OFFICE	MISS T. TAZELAAR

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, [11] with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	DE, A, 3415242 (FRAUNHOFER GESELLSCHAFT) 17 October 1985 see column 4, lines 3-61 --	1
A	Patent Abstracts of Japan, volume 7, no. 168 (P-212)(1313) 23 July 1983 & JP, A, 5875046 (NIPPON DENKI K.K.) 6 May 1983 see the abstract --	1
A	Patent Abstracts of Japan, volume 12, no. 390 (P-772)(3237), 18 October 1988 & JP, A, 63132139 (NIPPON SHEET GLASS) 4 June 1988 see the abstract ----	1

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. GB 9001332 SA 40115

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The members are as contained in the European Patent Office EDP file on 17/12/90
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO-A- 8907756	24-08-89	None	
DE-A- 3415242	17-10-85	None	

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82